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UNITED STATES ATOMIC ENERGY COMMISSION

CHAMBERS WORKS PROCESS FOR THE MANUFACTURE OF FLUORINE
CELL ELECTROLYTE (KF·2.3 HF) AT EAST AREA

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CHAMBERS WORKS PROCESS FOR THE MANUFACTURE OF FLUORINE
CELL ELECTROLYTE (KF·2.3 HF) AT EAST AREA*

HISTORICAL

This process is based upon experimental work carried out at Jackson Laboratory, and upon production at Semi-Works and Blue Products Area over a period of several months. This process supersedes that part of the Tentative Dye Works Process dated April 12, 1943 for the Manufacture of Fluorine, which relates to electrolyte manufacture.

PATENT STATUS

No investigation of patent status has been made.

PRINCIPLE

Anhydrous hydrofluoric acid (HF) is mixed with potassium acid fluoride (KHF₂) in such proportions that the resulting solution contains 43 to 44.5% HF. This material, which is liquid above 70°C, is used as the electrolyte in cells manufacturing gaseous fluorine (F₂).

YIELD

The yield per makeup charge is 5250 pounds of liquid electrolyte, corresponding to a yield of 98.7%, based upon the weights of KHF₂ and HF charged.

EQUIPMENT

501-14 - HF Tank Truck (D-59945).

Four-foot diameter by 10-foot long steel tank, capacity - 970 gallons; mounted on a 1 1/2 ton truck and equipped with a 2-inch standpipe, pressure gauge, 45-pound Pt rupture disc and 40-pound pop valve.

501 - HF Storage Tanks (2) (D-59905).

Eight-foot diameter by 30-foot long steel tanks on scales, capacity-11,588 gallons; equipped with a 2-inch standpipe, pressure gauge 45-pound Pt rupture disc and 40 pound pop valve.

1012 - HF Vaporizer (D-59867)

Four-foot diameter by 4-foot high steel tank on scales, bottom 1/3 jacketed, capacity - 430 gallons; equipped with 2-inch standpipe for

*Approved May 27, 1944.

feeding liquid HF, pressure gauge, jacket steam control system for maintaining constant tank pressure, 45-pound Pt rupture disc and 40-pound pop valve.

1006 - Electrolyte Makeup Tanks (2) (D-59915)

Four-foot, five-inch diameter by 2 foot, 9 1/2-inch high steam jacketed steel tanks, capacity - 600 gallons; equipped with a propeller agitator, brine-cooled condenser, standpipe for HF addition, electric probe, thermometer, pressure gauge, charging manhole, jacketed bottom outlet valve, 45-pound rupture disc, and 40-pound pop valve.

1003 - Electrolyte Standardizing Tanks (2) (D-59916)

Six-foot diameter by 5-inch high steam jacketed steel tanks on scales, capacity - 1250 gallons; equipped with flat-blade agitator, standpipe for HF addition, thermometer, pressure gauge, electric probe, jacketed bottom outlet valve, 45-pound Pt rupture disc, and 40-pound pop valve.

1001 - Electrolyte Storage Tanks (3) (D-59867)

Five-foot diameter by 13-inch long steel tanks, bottom third steam-jacketed, capacity - 1980 gallons; equipped with a jacketed bottom outlet valve, electric probe, rodding device, pressure gauge, thermometer, 45-pound Pt rupture disc, and 40-pound pop valve.

Auxiliary Equipment

Monorail system and chain fall for handling KHF_2 .

Air drier, filled with Silica gel, for supplying dry, 20-pound air to the system.

Four HF pumps, steel Worthington gear type, 25 gpm, for pumping HF to and from the HF storage tanks.

All electrolyte piping and valves are steam jacketed.

PROCEDURE

Materials	Mol. Wt.	Weight Used	Moles	M.R.	Wt.R	Used per 100-pound Product
Potassium Bifluoride (KHF_2)	78.10	4000	51.2	1.00	1.00	76.2
Hydrofluoric Acid (HF)	20.01	1317	65.8	1.29	0.33	25.1

HF Handling

HF is received by tank truck. The truck is weighed on the Chambers Works scales before and after loading to determine the amount received.

Pump 2700 to 2800 pounds of HF (enough for two charges of electrolyte) from the storage tank to the vaporizer. Raise the temperature of the HF with jacket steam until the pressure gauge reads 20 to 22 pounds.

Hazards

HF can cause painful burns in both the concentrated and dilute forms, and severe damage to the bones may result. A full acid suit is to be worn when lines containing HF are broken. Rubber gloves and a face mask are to be worn when operating HF valves.

In the event of accidental contact with HF, wash the affected parts immediately with copious quantities of cold water. Treatment with ice and/or magnesium oxide ointment is also in order. Carry the injured to the hospital immediately for treatment.

Electrolyte Makeup

Blow dry air through the makeup tank for an hour to remove HF fumes. Open the manhole cover and charge 4000 pounds of KHF_2 with the agitation on. Close the manhole, turn brine on the condenser, close the vent valve, and feed 1300 pounds of liquid HF into the tank. Maintain a temperature of 110 to 115°C (12-15 pound pressure) in the makeup tank by controlling the rate of HF addition. The pressure in the HF vaporizer must be kept above that in the makeup tank by admitting steam to the vaporizer jacket, or electrolyte will suck back into the vaporizer. If the agitation should stick, wait 5 minutes and start it again. Agitate for 1 hour after the HF addition is complete. Heat the charge to 90 to 120°C with jacket steam, and blow it to the standardizing tank.

Hazards:

KHF_2 reacts with water, liberating HF. Therefore, a full acid suit should be worn when charging the makeup tank.

The HF hazards are the same as under "HF Handling."

Standardization

Blow three makeup charges to one standardizing tank for standardization. Agitate the charge for approximately 1/2 hour and sample through the sampling nipple. Have the laboratory analyze for HF content by E.A. Method 216.70.

Liquid electrolyte can cause both HF and thermal burns. A full acid suit is to be worn when taking a sample.

SPECIFICATIONS

Anhydrous Hydrofluoric Acid, Special Distilled

Moisture	Not over 0.02%	J.L. Method 2676.05
SiF ₄	Not over 0.005%	E.A. Method 216.22
SO ₂	Not over 0.02%	E.A. Method 216.23

Potassium Acid Fluoride KHF₂

Purity	99% or higher	E.A. Method 216.50
Fluosilicates as K ₂ SiF ₆	Less than 1.0%	E.A. Method 216.54
Moisture	Less than 0.1%	E.A. Method 216.51
Sulfates as K ₂ SO ₄	Less than 0.05%	E.A. Method 216.53
Heavy Metals as Pb	Less than 0.6%	E.A. Method 216.55

Note: This specification is established on a temporary basis, since the only commercially available material requires this limit. It might subsequently be necessary to lower this figure.

Chlorides as KCl	Less than 0.1%	E.A. Method 216.52
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Electrolyte

HF Content	43.0 - 44.5%	E.A. Method 216.70
Moisture	Less than 0.05%	E.A. Method 216.71

Nitrogen

Chambers Works Specification 78-N.

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